

Inductively-Coupled-Plasma Atomic Emission Spectroscopy

Inductively-coupled-plasma atomic emission spectroscopy (ICP-AES) is used for quantitative determinations of elements at sample concentration levels ranging from major constituent to ppm. Simultaneous determination of up to 44 elements can be obtained on a single solution. A sequential instrument allows access to an even greater range of spectral lines and elemental methods.

Principle of Technique

Samples are introduced, generally as a nebulized solution, into an argon-supported, rf plasma discharge. Energetic conditions associated with the high-temperature plasma evaporate and atomize both solvent and solute. Plasma temperature is sufficient to electronically excite atoms of the analyte species; the subsequent radiative decay of these states produces photons characteristic of elements present. This light is focused, dispersed by means of a monochromator and detected photoelectrically. Multiple photomultiplier tubes located to intercept preset wavelengths are used by multichannel instruments to detect specific spectral lines. Line intensities are proportional to element concentrations in the sample.

Samples

Form. Liquids, usually aqueous solutions, can be analyzed as received or after dilution. Solids require dissolution.

Size. For liquid samples, 5 mL is required for the multichannel instrument, and about 1 mL/element for the scanning instrument. The minimum amount of analyte required varies by element but samples that provide µg quantities of analytes are typical.

Preparation. Solids must be dissolved in suitable medium, usually an aqueous acid solution. Extensive ashing may be required to prepare organic substances for analysis.

Limitations

Accuracy and precision of the method is 1 to 10%, depending on how well the background matrix is matched. Solids must be dissolved completely without introducing impurities. In specific situations, spectral interferences among elements in a sample may limit applicability. The method does not yield information on oxidation states or chemical speciation.

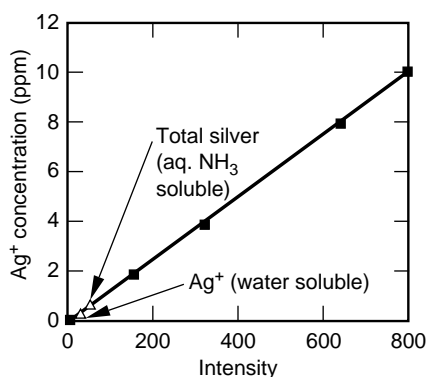
Estimated Analysis Time
Including instrument startup time, the analysis of a single sample generally requires 2 h. Quantitative determinations require 3 to 4 h for preparation of standards. Additional time may be needed for dissolution of difficult samples and for matrix separation, if required.

Examples of Applications

- Determination of leachable elements in geological systems.
- Determination of dopants in optical materials.
- Measurement of regulated metals in ground waters and hazardous waste.
- Quantitative analysis of alloys for major, minor, and trace components.

Metals Analysis						
Lawrence Livermore National Laboratory						
Environmental-Analytical Sciences						
Sample #: 94030165G-STLC2X				Sample type: STLC		
Lab book #D EAS ICP NTBK VOL 1 PG 15				Sample Matrix: AQUEOUS		
Date analyzed	Element	Analyst initials	Method number	Sample conc.	Units	MDL ^a
14-Oct-1994	Antimony	DGB	6010	ND	mg/L	0.1
14-Oct-1994	Arsenic	DGB	6010	ND	mg/L	0.2
14-Oct-1994	Barium	DGB	6010	0.29	mg/L	0.004
14-Oct-1994	Beryllium	DGB	6010	ND	mg/L	0.002
14-Oct-1994	Cadmium	DGB	6010	0.03	mg/L	0.01
14-Oct-1994	Chromium	DGB	6010	0.48	mg/L	0.01
14-Oct-1994	Cobalt	DGB	6010	0.12	mg/L	0.01
14-Oct-1994	Copper	DGB	6010	0.42	mg/L	0.01
14-Oct-1994	Lead	DGB	6010	0.1	mg/L	0.1
14-Oct-1994	Molybdenum	DGB	6010	0.02	mg/L	0.02
14-Oct-1994	Nickel	DGB	6010	0.64	mg/L	0.04
14-Oct-1994	^b Potassium	DGB	6010	48.	mg/L	0.4
14-Oct-1994	Selenium	DGB	6010	ND	mg/L	0.2
14-Oct-1994	Silver	DGB	6010	ND	mg/L	0.02
14-Oct-1994	Thallium	DGB	6010	ND	mg/L	0.2
14-Oct-1994	^b Uranium	DGB	6010	ND	mg/L	0.6
14-Oct-1994	Vanadium	DGB	6010	ND	mg/L	0.04
14-Oct-1994	Zinc	DGB	6010	4.2	mg/L	0.02
ND=Not Detected				MDL=Method Detection Limit		
a MDLs are estimated on MDLs measured in dilute standard solution						
b The concentration determined for this element is in support of the Department of Energy performance objective for identification and management of mixed waste. These data are relevant only for a sample that has been shown to be radioactive.						

This formal protocol includes both California and federally regulated elements.



ICP-AES analysis of a silver recovery effluent.

Capabilities of Related Techniques

DC-plasma optical emission spectroscopy is closely related, differing somewhat in the optics and type of plasma employed. It is also a multi-element technique with simultaneous detection. It has a higher spectral line resolution and generally higher detection limits than ICP-AES.

Inductively-coupled plasma mass spectrometry is also a multi-element technique. It has lower detection limits for many elements and provides isotopic information. The spe-

cific choice of technique often depends on the specific analyte and matrix elements involved.

DC-arc optical emission spectroscopy and x-ray fluorescence spectrometry may be applicable and can be used to analyze solids directly without dissolution. Quantitative analysis by these techniques is usually inferior.

Atomic absorption spectrometry (AAS) is more sensitive for some elements such as the alkali and alkali-earth elements. AAS, however, typically measures a single element at a time.

(H)	<div><div>Ag 3</div><div>← Element</div><div>← Typical detection limit using conventional solution nebulization (µg/L)</div></div>																(H)	(He)	
Li 5	Be 0.5											B 5	(C)	(N)	(O)	(F)	(Ne)		
Na 2	Mg 0.5											Al 20	Si 15	P 50	S 5	(Cl)	(Ar)		
K 60	Ca 0.5	Sc 1	Ti 2	V 2	Cr 5	Mn 1	Fe 5	Co 7	Ni 7	Cu 2	Zn 2	Ga 30	Ge 50	As 45	Se 50	(Br)	(Kr)		
Rb 500	Sr 1	Y 2	Zr 3	Nb 20	Mo 5	(Tc)	Ru 50	Rh 20	Pd 40	Ag 3	Cd 2	In 40	Sn 25	Sb 50	Te 60	(I)	(Xe)		
(Cs)	Ba 1	La 5	Hf 10	Ta 20	W 30	Re 8	Os 20	Ir 20	Pt 20	Au 10	Hg 30	Tl 50	Pb 25	Bi 30	(Po)	(At)	(Rn)		
(Fr)	(Ra)	(Ac)	(Rf)	(Ha)															
Ce 30	Pr 20	Nd 10	(Pm)	Sm 10	Eu 2	Gd 10	Tb 10	Dy 3	Ho 3	Er 10	Tm 3	Yb 1	Lu 1						
Th 30	(Pa)	U 60	(Np)	(Pu)	(Am)	(Cm)	(Bk)	(Cf)	(Es)	(Fm)	(Md)	(No)	(Lr)						

ICP-AES detection limits ($\mu\text{g/l}$) under optimum conditions using conventional aqueous solution nebulization are noted below each element. Quantitative analysis limits are generally 10 times the detection limit. Highlighted elements have especially good sensitivity. Those in parentheses are not usually determined by ICP-AES due to interferences, instability, the lack of good spectral lines or suitable standards, or other factors.